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MANUFACTURING METHOD OF FIBER REINFORCED THERMOSETTING RESIN MOLDING

[Seni kyoka kokasei jushi seikeihin no seizoho]

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Claim

A manufacturing method of fiber reinforced thermosetting resin molding characterized by the following facts: when the molding is manufactured from the fiber reinforcing material and thermosetting resin, the fiber reinforcing material in mat shape, cloth shape and/or roving shape is laminated in the dies; the dies are erected so that the laminating direction becomes horizontal; while the interior of the dies is in a reduced-pressure state, the thermosetting resin is fed to impregnate from the bottom portion of the fiber reinforcing material inside the dies; after the entirety of the fiber reinforcing material is impregnated, the interior of the dies is restored to ambient pressure, followed by heating under ambient pressure.

Detailed explanation of the invention

The fiber reinforced thermosetting resin molding has excellent mechanical properties, so that it is widely used in industry, such as in various types of spacers, base plates for printed circuit boards, etc. However, when it is used as the electrical insulating members in heavy electric machines, etc., in addition to good electrical properties, it also should have high homogeneity in structure and high reliability free of any voids caused by enclosed bubbles, cracks, etc.

In the prior art, there are various manufacturing methods to mold the fiber reinforced thermosetting resin moldings. However, they have disadvantages. For example, there is the following method in the prior art: the fiber reinforcing material is coated and impregnated with the thermosetting resin (hereinafter to be referred to as resin) and is half-cured to form the so-called prepreg sheets. The prepreg sheets are then laminated and heated under pressure to form a laminate plate. However, this method also has some problems, such as [insufficient] shelf life of the prepreg sheets, as well as insufficient prevention of enclosed bubbles.

As another method in the prior art, the fiber reinforcing material is laminated in dies, and after the dies are inserted in a vacuum tank, the overall vacuum tank is evacuated, and the de-foamed resin impregnates from the periphery of the fiber reinforcing material. Then, the pressure is brought back to the ambient pressure, followed by heating for curing under the ambient pressure to form a laminate plate. However, in this method, there is the disadvantage that bubbles are left in the central portion of the laminate plate.

As a method for improving said method, after the pressure is brought back to ambient pressure, curing is performed in a pressurized state to form the laminate plate. When the pressure is high, the bubbles in the central portion become dissolved and are eliminated in the epoxy resin. However, in this method, when the pressure application medium is a gas, the heat transfer efficiency is very low, so that it is difficult to heat the dies in the interior. On the other hand, when a liquid pressure application medium is adopted, it may be mixed with the epoxy resin, or the pressure application medium may stick on the dies. In both cases, the operation takes a long time and requires many man-hours. This is undesirable.

The purpose of the present invention is to solve the aforementioned problems of the prior art by providing a simple and cost-effective manufacturing method of the fiber reinforced thermosetting resin molding with excellent mechanical properties and electrical properties and free of bubbles and cracks, etc.

The present invention provides a manufacturing method of a fiber reinforced thermosetting resin molding with excellent mechanical properties and electrical properties. More specifically, the present invention provides a manufacturing method of fiber reinforced thermosetting resin molding characterized by the following facts: when the molding is manufactured from the fiber reinforcing material and thermosetting resin, the fiber reinforcing material in mat shape, cloth shape and/or roving shape is laminated in the dies; the dies are erected so that the laminating direction becomes horizontal;

while the interior of the dies is in a reduced-pressure state, the thermosetting resin is fed to impregnate from the bottom portion of the fiber reinforcing material inside the dies; after the entirety of the fiber reinforcing material is impregnated, the interior of the dies is restored to ambient pressure, followed by heating under ambient pressure.

The present inventors have observed the following facts: when said fiber reinforcing material in mat shape, cloth shape and/or roving shape is laminated in dies, and a thermosetting resin is injected into the dies so that the fiber reinforcing material is impregnated with the thermosetting resin, the air or other air contained in the fiber reinforcing material should be substituted, so that no bubbles are enclosed; for this purpose, the interior of the dies where the fiber reinforcing material is laminated has the pressure reduced so that the viscosity of the gas contained in the fiber reinforcing material is decreased, the relative viscosity of the gas contained in the fiber reinforcing material with respect to the viscosity of the resin is decreased, so that exhaustion of the gas is facilitated, and at the same time, flow of the resin is adjusted so that the fiber reinforcing material inside the dies is impregnated with the thermosetting resin from its bottom portion, and the gas contained in the fiber reinforcing material is exhausted.

According to the manufacturing method of the present invention, it is possible to manufacture the fiber reinforced thermosetting resin molding with excellent mechanical properties and electrical properties, free of bubbles, cracks, etc., easily and at a low cost.

Here, the fiber reinforcing material is prepared as one type or a combination of two or more types of the following fibers selected according to the specific purpose: glass fibers, carbon fibers, asbestos and other inorganic fibers, cotton, nylon, and other organic fibers, etc. The forms of the fiber reinforcing material include mat shape, cloth shape and/or roving shape, either isotropic or anisotropic.

The roving-shape reinforcing material can be used in manufacturing unidirectionally reinforced resin laminate plates. In this case, the roving-shape fiber reinforcing material should be aligned beforehand using the FW method or the like.

The fiber reinforcing material is cut into pieces with appropriate dimensions and fiber content, and the pieces of the fiber reinforcing material are laminated in the dies. The dies are then erected so that the laminating direction becomes horizontal. The resin flows very quickly in the fiber direction in the fiber reinforcing material, while it flows very slowly in the direction perpendicular to the fiber direction. Consequently, according to the method of the present invention, even when molding is performed without specifically applying a pressure, impregnation can still be well realized, and a homogeneous molding still can be obtained. In this case, it is preferred that a space be set between the lower end of the fiber reinforcing material and the dies for adjusting the flow of the thermosetting resin.

As the thermosetting resin, in consideration of the mechanical properties and electrical properties, one may use a solvent-free liquid resin, such as epoxy resin, unsaturated polyester resin as well as their modified resins, etc. Also, corresponding to the specific purpose of application, one may add appropriate types of curing agents, curing accelerating agents, plasticity improving agents, fillers, pigments, etc., in said solvent-free liquid resin.

According to the present invention, while the dies are erected so that the laminating direction of the fiber reinforcing material is in the horizontal direction and the interior of the dies is in a reduced-pressure state, the resin is made to impregnate the fiber reinforcing material from its bottom portion. Here, the interior of the dies may be evacuated after the dies are erected, or the dies may be erected after the interior of the dies has been evacuated. It is preferred that the reduced pressure in the interior of the dies be 10 torr or lower. When the thermosetting resin is impregnated from the bottom portion of the fiber reinforcing material inside the dies, it is preferred that the injection pressure in this

case be adjusted corresponding to the type, form and fiber content of the fiber reinforcing material, the viscosity of the resin, the pressure loss of the pipeline, the dimensions and shape of the dies, etc., such that the injected resin flow rate is appropriate. Also, it is preferred that once the entire fiber reinforcing material is impregnated with the thermosetting resin, injection of the resin be stopped, and the dies be set such that their interior is in a sealed state, as this can further ensure a complete removal of bubbles.

As a preferable scheme, a vacuum pump is connected to the top portion of the dies as a port different from the injecting port to perform continuous evacuation while injection is being performed. In this case, the bubbles can be fully removed.

The following scheme may also be adopted according to the present invention: in order to impregnate the fiber reinforcing material with the thermosetting resin from the bottom portion of the fiber reinforcing material, the resin is injected into the dies through an injecting port set on the bottom portion of the dies, so that the thermosetting resin is impregnated from the bottom portion of the fiber reinforcing material inside the dies. Also, the following scheme may be adopted: the resin is injected into the dies through an injecting port formed on the upper portion of the sidewall of the dies, and after the resin is transferred by a pipe to the bottom portion of the dies, the resin is impregnated from the bottom portion of the fiber reinforcing material inside the dies. There is no specific restriction on the position of the dies for injecting the resin into the dies. It is preferred that the resin to be impregnated in the fiber reinforcing material be defoamed beforehand before injecting into the dies, as this can prevent enclosing of the bubbles.

After impregnation, the pressure in the dies is brought back to ambient pressure. In this case, however, attention should be paid to the fact that if the pressure increasing rate is too high, the gas will backflow to the fiber reinforcing material. Then, under ambient pressure, the dies are heated for thermosetting, forming a fiber reinforced thermosetting resin molding.

In the following, an explanation will be given in more detail regarding the present invention with reference to application examples.

Application Example 1

As said fiber reinforcing material, glass cloth (commercial name WE-35DBX, product of Nitto Spinning Co., Ltd., unit weight of 310 g/m^2) was cut into 80 pieces measuring 1 m square. These pieces of glass cloth were laminated in a set of dies. As shown in Figures 1 and 2, dies (1) with said pieces of glass cloth (2) laminated in the dies were then erected so that the laminating direction of the glass cloth became horizontal. In order to prevent falling of the glass cloth without hindering the flow of the resin, fiber reinforcing material fall preventing block (5) with grooves cut on the lower side was set on the lower end of the glass cloth, and the interior of the dies was evacuated from exhausting port (4) in 5 min to a vacuum degree of 3 torr or less. Then, a pre-blended defoamed and preheated (at 60°C) liquid epoxy resin composition (a mixture of 100 parts by weight of epoxy resin: commercial name CY-225, product of Ciba Geigy Corp. and 80 parts by weight of modified acid anhydride curing agent: commercial name of HY-225, product of Ciba Geigy Corp.) was injected for about 10 min through injecting port (3) under a pressure of 50 torr until the resin over-flowed, followed by allowing to stand for 10 min. Then, after injection for another 2 min, it was set for 5 min, and it was brought back to ambient pressure in 10 min. The dies were then carried into a desiccator at 100°C, and it was heated for 2 h for thermosetting. Then, the molding was removed from the disassembled dies.

As listed in Table 1, for the obtained fiber reinforced thermosetting resin molding, excellent electrical properties and mechanical properties were realized, and it was found to be free of bubbles and cracks.

Comparative Example 1

As the fiber reinforcing material, glass cloth (2) (commercial name WE-35DBX, product of Nitto Spinning Co., Ltd., unit weight of 310 g/m^2) was cut into 80 pieces measuring 1 m square. These pieces of glass cloth were laminated in a set of dies (1). As shown in Figure 3, the dies with said pieces of glass cloth (2) laminated in them were set as is so that the laminating direction of the glass cloth was in the vertical direction. The interior of the dies (1) was evacuated from exhausting port (4) in 5 min to a vacuum degree of 3 torr or less. Then, a pre-blended defoamed and preheated (at 60°C) liquid epoxy resin composition (a mixture of 100 parts by weight of epoxy resin: commercial name CY-225, product of Ciba Geigy Corp. and 80 parts by weight of modified acid anhydride curing agent: commercial name of HY-225, product of Ciba Geigy Corp.) was injected for about 2 min through injecting port (3) under a pressure of 50 torr until the resin overflowed, followed by allowing to stand for 10 min. Then, after injection and standing were performed repeatedly 5 times, it was brought back to ambient pressure in 10 min. The dies were then carried into a desiccator at 100°C, and it was heated for 2 h for thermosetting. Then, the molding was removed from the disassembled dies. As listed in Table 1, for the fiber reinforced thermosetting resin molding formed in this case, fine bubbles appeared over the entire surface, the mechanical properties were degraded somehow, and the electrical properties degraded significantly. This is undesirable.

TABLE 1

項目	実施例1	比較例1
外観	○	×
クラック	○	○
表面平滑性	○	×
絶縁破壊電圧 (kV/2mm)	32	26
曲げ強さ (kgf/mm²)	42	33
ガラス繊維重量含有率 (%)	60	59
空隙率 (%)	0	1〜3

Key: 1 Item

2 Application Example 1

Comparative Example 1

3 Bubbles

Cracks

Surface smoothness

Voltage rating through layers (kV/2 mm)

Flexural strength (kgf/mm^2)

Content of glass fibers (%)

Porosity (%)

Here, for evaluating the bubbles and cracks, the laminate plate was cut, and the appearance of the cross section was visually observed to evaluate with the following grades:

O: No bubbles or cracks at all

X: There are bubbles and cracks

On the other hand, for the surface smoothness, the bumps/dips of the surface were measured and evaluation was made with the following grades:

O: smaller than 0.2 mm or smaller

X: 0.2 mm or larger

For the voltage rating through layers, the flexural strength and the porosity, tests were performed according to JIS K6911. The content by weight of the glass fibers was determined using the incineration method.

According to the manufacturing method of the present invention, replacement of the gas between portions of the fiber reinforcing material with the thermosetting resin can be performed perfectly and easily, and although curing is performed under ambient pressure, bubbles are still not generated, and the fiber reinforced thermosetting resin molding with excellent electrical properties and mechanical properties can be manufactured at a low cost.

Brief description of the figures

Figures 1 and 2 illustrate the dies and laminating state of the fiber reinforcing material in Application Example 1. Figure 3 is a diagram illustrating the dies and the laminating state of the fiber reinforcing material in Comparative Example 1.

Explanation of symbols

1 Dies

2 Fiber reinforcing material

3 Injecting port

4 Exhausting port

5 Fiber reinforcing material fall preventing block

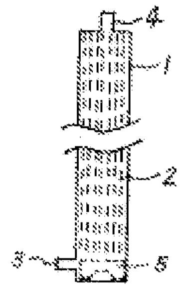

Figure 1

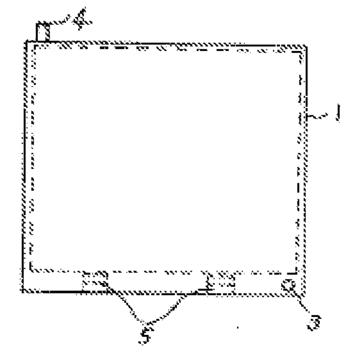

Figure 2

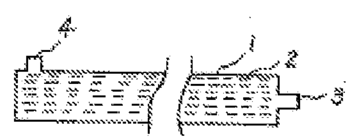

Figure 3